

10670409-isocyanate

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10670409-isocyanate

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FILE COVERS 1907 - 21 Jun 2004 VOL 140 ISS 26  
FILE LAST UPDATED: 20 Jun 2004 (20040620/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s diisocyanate

L1 43031 DIISOCYANATE

→ product

=> s diamine

L2 39888 DIAMINE

→ reactant

=> s l1 and l2

L3 1871 L1 AND L2

←

=> s phosgene

L4 12338 PHOSGENE

← reactant

=> s l4 and l3

(L5) 64 L4 AND L3

←

=> s "hydrogen chloride"

823591 "HYDROGEN"

991591 "CHLORIDE"

(L6) 26359 "HYDROGEN CHLORIDE"

("HYDROGEN" (W) "CHLORIDE")

← reactant

=> s l6 and l5

(L7) 5 L6 AND L5

5 reference

=> s phosgenation

L8 1238 PHOSGENATION

← general Rxn name

=> s l8 and l5

L9 31 L8 AND L5

=> s l9 or l5 or l7

L10 64 L9 OR L5 OR L7

total with Rxn + ~~Product~~ Product

=> s spray

L11 110287 SPRAY

in nozzle

=> s l11 and l10

L12 0 L11 AND L10

Required in improvement

=> s "spray nozzles"

110287 "SPRAY"

24235 "NOZZLES"

L13 1621 "SPRAY NOZZLES"

("SPRAY" (W) "NOZZLES")

10670409-isocyanate

=> s l13 and l10

L14 0 L13 AND L10

=> s l13 and l3

L15 0 L13 AND L3

=> s bib abs 1-31 l9

MISSING OPERATOR 1-31 L9

The search profile that was entered contains terms or  
nested terms that are not separated by a logical operator.

=> d bib abs 1-31 l9

L9 ANSWER 1 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2004:261001 CAPLUS

DN 140:287841

TI Process for the preparation of polyisocyanates of the diphenylmethane series by **phosgenation** of nonneutralized polyamines of the diphenylmethane series which are prepared by the reaction of aniline with aqueous formaldehyde in the presence of HCl

IN Koch, Daniel; Pirkel, Hans-Georg; Hagen, Torsten; Wershofen, Stefan

PA Bayer Materials science A.-G., Germany

SO Eur. Pat. Appl., 7 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1403242	A1	20040331	EP 2003-20534	20030917
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
	DE 10245703	A1	20040401	DE 2002-10245703	20020930
	US 2004092701	A1	20040513	US 2003-672440	20030926
	JP 2004123746	A2	20040422	JP 2003-341556	20030930
PRAI	DE 2002-10245703	A	20020930		
AB	Polyisocyanates of the diphenylmethane series (e.g., MDI) are prepared by <b>phosgenation</b> of nonneutralized polyamines (e.g., the corresponding <b>diamine</b> of MDI) of the diphenylmethane series which are prepared by the reaction of aniline with aqueous formaldehyde in the presence of HCl, where distillation of the water from the formed <b>diamine</b> before <b>phosgenation</b> is a process step.				

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 2 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2004:159009 CAPLUS

DN 140:199902

TI Process for the manufacture of aromatic diisocyanates via the gas-phase **phosgenation** of aromatic diamines under moderate pressures

IN Woelfert, Andreas; Mueller, Christian; Stroeder, Eckhard; Weber, Markus; Pfeffinger, Joachim; Knoesche, Carsten

PA BASF A.-G., Germany

SO Ger. Offen., 7 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 10238995	A1	20040226	DE 2002-10238995	20020820
	WO 2004026813	A1	20040401	WO 2003-EP8108	20030724

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W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRAI DE 2002-10238995 A 20020820

AB Aromatic diisocyanates (e.g., TDI) are prepared in high yield and selectivity the gas-phase reaction of **phosgene** with aromatic diamines (e.g., toluenediamine) under moderate pressures of >3 bars and <20 bars. A process flow diagram is presented.

L9 ANSWER 3 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:950981 CAPLUS

DN 140:5423

TI **Phosgenation** method and reactors for producing isocyanates from **phosgene** and primary amines

IN Woelfert, Andreas; Mueller, Christian; Stroefer, Eckhard; Brodhagen, Andreas

PA Basf Aktiengesellschaft, Germany

SO PCT Int. Appl., 18 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003099770	A1	20031204	WO 2003-EP5232	20030519
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				

DE 10222968 A1 20031204 DE 2002-10222968 20020523

PRAI DE 2002-10222968 A 20020523

AB A continuous method for producing isocyanates (e.g., TDI) by reacting primary amines (e.g., toluenediamines) with **phosgene** is described in which the reaction is carried out in a cascade of at least two tubular reactors and, after each reactor, the gas phase resulting during the reaction is separated in a phase separator, and only the liquid

phase is fed to the next reactor or to the product purification. In addition, the reaction volume of the first tubular reactor is equal to only a fraction of the total reaction volume.

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:910219 CAPLUS

DN 139:381885

TI **Phosgenation** process and reactor for the gas-phase preparation

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of isocyanates from their corresponding amines  
IN Jenne, Marc; Herold, Heiko; Friedrich, Martin; Stutz, Herbert  
PA Bayer Aktiengesellschaft, Germany  
SO Eur. Pat. Appl., 6 pp.  
CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1362847	A2	20031119	EP 2003-10096	20030505
	EP 1362847	A3	20040204		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
	DE 10222023	A1	20031127	DE 2002-10222023	20020517
	US 2003216597	A1	20031120	US 2003-437509	20030514
	CN 1458150	A	20031126	CN 2003-136025	20030516
	JP 2004067669	A2	20040304	JP 2003-138482	20030516
PRAI	DE 2002-10222023	A	20020517		
OS	MARPAT 139:381885				
AB	A tubular <b>phosgenation</b> reactor, which exhibits reduced temperature fluctuations and thus has reduced polymeric buildup, is described as is a process for the gas-phase <b>phosgenation</b> of diamines (e.g., hexamethylenediamine) or triamines into their corresponding isocyanates (e.g., hexamethylenediisocyanate).				

L9 ANSWER 5 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:892453 CAPLUS

DN 139:382379

TI Manufacture of tetralin isocyanates as monomers for polyurethanes

IN Adkins, Rick L.; Parsons, Harold R.

PA Bayer Polymers, LLC, USA

SO U.S. Pat. Appl. Publ., 3 pp.

CODEN: USXXCO

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003212160	A1	20031113	US 2002-142289	20020509
	US 6750367	B2	20040615		
	WO 2003095419	A1	20031120	WO 2003-US14011	20030505
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
PRAI	US 2002-142289	A	20020509		

AB Novel aromatic isocyanates, useful as isocyanate components in the production of

flexible polyurethane foam, are obtained by **phosgenation** of diaminotetralin in 50/50 diglyme/1,2-dichlorobenzene mixture The latter **diamine** was prepared by nitration of tetralin and reduction of dinitrotetralin. Thus, polyurethane foam manufactured from tetralin **diisocyanate** 100, glycerol/propylene glycol-based polyol 100, H<sub>2</sub>O 4.40, L620 (silicone surfactant) 0.8 and Polycat 70 catalyst 1.0 part had

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d. 1.31 lb/ft<sup>3</sup>, tensile strength 17.3 lb/in<sup>2</sup>, elongation 208% and tear strength 2.13 lb/in, vs. 1.30, 17.7, 168 and 1.72, resp., for polyurethane foam manufactured with 100 parts TDI instead of tetralin **diisocyanate**

L9 ANSWER 6 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:434519 CAPLUS

DN 139:22615

TI Production of isocyanates in the gaseous phase

IN Woelfert, Andreas; Mueller, Christian; Stroeyer, Eckhard; Pfeffinger, Joachim; Weber, Markus; Knoesche, Carsten

PA BASF Aktiengesellschaft, Germany

SO PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2003045900	A1	20030605	WO 2002-EP12930	20021119
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
	RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
	DE 10158160	A1	20030612	DE 2001-10158160	20011128
PRAI	DE 2001-10158160	A	20011128		
AB	The invention relates to a method for producing diisocyanates by reacting primary diamines with <b>phosgene</b> in the gaseous phase. Said method is characterized in that the reaction of <b>diamine</b> and <b>phosgene</b> occurs in a reaction channel, the internal dimensions of which have a width/height ratio of at least 2/1. With these dimensions the reaction chamber is useful for a longer period of time before it is necessary to clean the chamber of solid precipitate				
RE.CNT	2	THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD			
		ALL CITATIONS AVAILABLE IN THE RE FORMAT			

L9 ANSWER 7 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:40165 CAPLUS

DN 138:90237

TI Process and apparatus for the preparation of (cyclo)aliphatic di- and triisocyanates by the gas-phase **phosgenation** of the corresponding diamines and triamines

IN Leimkuehler, Hans-Joachim; Stutz, Herbert; Schmidt, Helmut

PA Bayer AG, Germany

SO Eur. Pat. Appl., 7 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1275640	A1	20030115	EP 2002-14039	20020628
	R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR			
	DE 10133728	A1	20030123	DE 2001-10133728	20010711

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US	2003069441	A1	20030410	US	2002-190262	20020703
US	6706913	B2	20040316			
JP	2003096043	A2	20030403	JP	2002-195908	20020704
CN	1396153	A	20030212	CN	2002-140950	20020711
PRAI	DE 2001-10133728	A	20010711			

OS MARPAT 138:90237

AB (cyclo)aliphatic di- (e.g., 1,6-diisocyanatohexane) and triisocyanates are prepared in high yield and selectivity by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,6-diaminohexane) and triamines at 200-600° where the **phosgene** is injected into the vaporized amine stream at a point in the reactor where the reactor internal diameter is narrowed and then re-expands past the point of the **phosgenation** reaction. An apparatus diagram is presented.

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 8 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:40164 CAPLUS

DN 138:90236

TI Process and apparatus for the preparation of (cyclo)aliphatic di- and triisocyanates by the gas-phase **phosgenation** of the corresponding diamines and triamines

IN Leimkuehler, Hans-Joachim; Stutz, Herbert; Leuckel, Wolfgang

PA Bayer Aktiengesellschaft, Germany

SO Eur. Pat. Appl., 9 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1275639	A1	20030115	EP 2002-14038	20020628
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
DE 10133729	A1	20030123	DE 2001-10133729	20010711
US 2003013909	A1	20030116	US 2002-190265	20020703
JP 2003104948	A2	20030409	JP 2002-195902	20020704
CN 1396152	A	20030212	CN 2002-140947	20020711

PRAI DE 2001-10133729 A 20010711

OS MARPAT 138:90236

AB (cyclo)aliphatic di- (e.g., 1,6-diisocyanatohexane) and triisocyanates are prepared in high yield and selectivity by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,6-diaminohexane) and triamines at 200-600° where the **phosgene** is injected into the gaseous amine stream at a point in the reactor where the reactor internal diameter is narrowed and then re-expands past the point of the **phosgenation** reaction. An apparatus diagram is presented.

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 9 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:488630 CAPLUS

DN 135:61735

TI Method for making aliphatic diisocyanates which uses supercritical extraction of the process waste stream

IN Sommer, Alexa B.; Wittig, Mary Ann; Hortelano, Edwin R.; Yeske, Philip E.; Ciebien, Jane F.

PA Bayer Corporation, USA

SO Eur. Pat. Appl., 14 pp.

CODEN: EPXXDW

DT Patent

LA English

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FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1113003	A1	20010704	EP 2000-127231	20001215
	EP 1113003	B1	20030820		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	US 6307096	B1	20011023	US 1999-473097	19991228
	AT 247636	E	20030915	AT 2000-127231	20001215
PRAI	US 1999-473097	A	19991228		
AB	A method for making an aliphatic <b>diisocyanate</b> (e.g., hexamethylene <b>diisocyanate</b> ) is described comprising: (a) phosgenating an aliphatic <b>diamine</b> (e.g., 1,6-diaminohexane) in the presence of an inert solvent or gas to form a crude reaction mixture; (b) distilling the crude reaction mixture to form an aliphatic <b>diisocyanate</b> production stream and an aliphatic <b>diisocyanate</b> waste stream; (c) introducing the aliphatic <b>diisocyanate</b> waste stream to a chamber and placing the waste stream under supercrit. fluid conditions sufficient to dissolve an appreciable amount of the aliphatic <b>diisocyanate</b> component in the supercrit. fluid (e.g., carbon dioxide); (d) separating the dissolved aliphatic <b>diisocyanate</b> component from the waste stream, where the remaining waste stream is a supercritically-purged aliphatic <b>diisocyanate</b> waste stream; and (e) lowering the pressure sufficiently to precipitate the aliphatic <b>diisocyanate</b> component.				

RE.CNT 4      THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9      ANSWER 10 OF 31    CAPLUS    COPYRIGHT 2004 ACS on STN  
AN      2001:165790    CAPLUS  
DN      134:207403  
TI      Improved procedure for the production of mono- and oligoisocyanates by the **phosgenation** of primary amines in the presence of catalytic amounts of monoisocyanates  
IN      Stamm, Armin; Kneuper, Heinz-josef; Thil, Lucien; Henkelmann, Jochem  
PA      BASF AG, Germany  
SO      Ger. Offen., 8 pp.  
CODEN: GWXXBX  
DT      Patent  
LA      German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19942299	A1	20010308	DE 1999-19942299	19990904
	WO 2001017951	A1	20010315	WO 2000-EP8221	20000823
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	EP 1208082	A1	20020529	EP 2000-951530	20000823
	EP 1208082	B1	20040407		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
	AT 263752	E	20040415	AT 2000-951530	20000823
	US 6683204	B1	20040127	US 2002-70393	20020304
PRAI	DE 1999-19942299	A	19990904		
	WO 2000-EP8221	W	20000823		
OS	CASREACT 134:207403; MARPAT 134:207403				



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AB Aliphatic, cycloaliph., araliph. [e.g., R-(+)-phenylethyl isocyanate], or aromatic mono- and oligoisocyanates are prepared in high yield and selectivity by the **phosgenation** of the appropriate primary amines [e.g., R-(+)-phenylethylamine] at atmospheric pressure with **phosgene** in the presence of catalytic amts. of monoisocyanates (e.g., Bu isocyanate) in an inert solvent (e.g., chlorobenzene).

L9 ANSWER 11 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:629973 CAPLUS

DN 129:231150

TI Process for the preparation of diisocyanates of light color by the **phosgenation** of diamines treated with inorganic solids having Lewis and/or Bronsted acid centers

IN Kraus, Rupert; Reif, Martin; Bruchmann, Bernd; Tesch, Helmut

PA BASF A.-G., Germany

SO Eur. Pat. Appl., 5 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 866057	A2	19980923	EP 1998-103997	19980306
	EP 866057	A3	20020814		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	DE 19711447	A1	19980924	DE 1997-19711447	19970319
	CA 2229835	AA	19980919	CA 1998-2229835	19980318
	US 5872278	A	19990216	US 1998-40673	19980318
	CN 1197793	A	19981104	CN 1998-108727	19980319
	CN 1070475	B	20010905		
	JP 10306068	A2	19981117	JP 1998-70194	19980319
PRAI	DE 1997-19711447	A	19970319		

AB Diisocyanates (e.g., MDI) having little discoloration, suitable as monomers for the manufacture of polyurethanes (no data), are prepared by the treatment of diamines [e.g., 1,1'-methylenebis(4-aminobenzene)] with inorg. solids which contain Lewis and/or Bronsted acid centers (e.g., mol. sieves) followed by **phosgenation** of the treated diamines.

L9 ANSWER 12 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1996:248012 CAPLUS

DN 124:261969

TI Continuous manufacture of aromatic diisocyanates

IN Bestiuc, Ioan; Caraculacu, Adrian; Idriceanu, Silvia; Buruiana, Tinca; Kelemen, Zoltan; Pogor, Constantin

PA Inst. de Chimie Macromoleculara "Petru Poni", Iasi, Rom.

SO Rom., 6 pp.

CODEN: RUXXA3

DT Patent

LA Romanian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	RO 104788	B1	19940920	RO 1989-143106	19891211
PRAI	RO 1989-143106		19891211		

AB Aromatic diisocyanates are manufacture by intimately contacting a solution of 5-20%

aromatic diamines in a solvent selected from hydrocarbons, chloride compds., esters, or ethers with COCl<sub>2</sub> at COCl<sub>2</sub>-**diamine** mol ratio >2.5 using a pump mounted on the shaft of a stirrer, with recirculation of a HCl-COCl<sub>2</sub> gas mixture from the upper part of the reactor through the reaction mixture This method provides product with low concentration of

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hydrolyzable Cl and eliminates the requirement of 1st forming hydrochlorides of the diamines.

L9 ANSWER 13 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:994743 CAPLUS  
DN 124:86379  
TI Process for the preparation of diisocyanates by the **phosgenation** of diamines  
IN Bischoff, Eric; Breidenbach, Peter; Dahmer, Juergen; Flink, Andreas; Molnar, Attila; Stutz, Herbert  
PA Bayer A.-G., Germany  
SO Eur. Pat. Appl., 6 pp.  
CODEN: EPXXDW  
DT Patent  
LA German  
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 676392	A1	19951011	EP 1995-104637	19950329
	EP 676392	B1	20010620		
	R: BE, DE, ES, FR, GB, IT, NL				
	DE 4412327	A1	19951012	DE 1994-4412327	19940411
	EP 1078918	A1	20010228	EP 2000-202871	19950329
	EP 1078918	B1	20021211		
	R: BE, DE, ES, FR, GB, IT, NL				
	ES 2159579	T3	20011016	ES 1995-104637	19950329
	ES 2188472	T3	20030701	ES 2000-202871	19950329
	US 5516935	A	19960514	US 1995-413646	19950330
	CA 2146522	AA	19951012	CA 1995-2146522	19950406
	JP 07278089	A2	19951024	JP 1995-107109	19950407
PRAI	DE 1994-4412327	A	19940411		
	EP 1995-104637	A3	19950329		

AB Diisocyanates (e.g., 1,3-pentane **diisocyanate**) are prepared by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,3-diaminopentane) in which: gaseous diamines, if necessary diluted with inert gas or inert-solvent vapors, and **phosgene**, are sep. heated to 200-600° and continuously brought together in a non-stirred cylindrical reactor heated to 200-600° by maintaining a turbulent stream so they react; the gas mixture continuously leaving the reactor with the help of an inert liquid solvent, that is kept at a temperature above the decomposition temperature of the carbamic acid chloride corresponding to the **diamine**, is condensed to give a solution of the **diisocyanate** in this solvent; and the desired **diisocyanate** dissolved in the inert solvent is recovered by distillation

L9 ANSWER 14 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:992543 CAPLUS  
DN 124:88109  
TI Preparation of aliphatic polyisocyanates from polyamines and **phosgene**  
PA Mitsui Toatsu Chemicals, Inc., Japan  
SO Ger. Offen., 11 pp.  
CODEN: GWXXBX  
DT Patent  
LA German  
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19510259	A1	19950928	DE 1995-19510259	19950321
	DE 19510259	C2	19970904		
	JP 07309827	A2	19951128	JP 1995-42956	19950302
	JP 3201921	B2	20010827		

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US 5523467 A 19960604 US 1995-401807 19950310  
CN 1125718 A 19960703 CN 1995-104546 19950322  
CN 1062857 B 20010307  
PRAI JP 1994-50082 A 19940322

AB In the conversion of an aliph polyamine to the polyisocyanate (e.g., m-xylylenediamine to m-xylylene **diisocyanate**) in an inert liquid medium, an inert gas is added to the reactor during the reaction to increase the yield of polyisocyanate and reduce the amount of **phosgene** required.

L9 ANSWER 15 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:103343 CAPLUS  
DN 122:188201  
TI 1,3-Pentane **diisocyanate**  
AU Sheridan, Robert E.  
CS E. I. Du Pont and Co., USA  
SO Research Disclosure (1994), 362, 306 (No. 36220)  
CODEN: RSDSBB; ISSN: 0374-4353  
DT Journal; Patent  
LA English

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	---	-----	-----	-----
PI RD 362020		19940610		
PRAI RD 1994-362020		19940610		
AB Direct hot <b>phosgenation</b> of 1,3-diaminopentane at 125° in m-chlorobenzene resulted in 30% yield of 1,3-pentane <b>diisocyanate</b> . Subsequent distillation after addition of di-ortho-xylylethane resulted in 34% recovery (10% based on starting <b>diamine</b> ) of 1,3-pentane <b>diisocyanate</b> which was 82-85% pure. The product was characterized by IR and mass spectroscopy.				

L9 ANSWER 16 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1994:701572 CAPLUS  
DN 121:301572  
TI Manufacture of diphenylmethane 4,4'-**diisocyanate**  
IN Botezatu, Petru; Miron, Ada Margareta; Olanescu, Emil; Stoica, Dumitru; Teodorescu, Dan Cezar  
PA Combinatul de Fibre Sintetice, Savinesti, Rom.  
SO Rom., 3 pp.  
CODEN: RUXXA3  
DT Patent  
LA Romanian  
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI RO 103439	B1	19921123	RO 1989-138956	19890329
PRAI RO 1989-138956		19890329		
AB Title compound, useful for the manufacture of polyurethanes (no data), is prepared by <b>phosgenation</b> of the dihydrochloride of isomer mixts. containing 96-98% 4,4'-diaminodiphenylmethane 8 h in polyalkylbenzene at 140-160° and CO2Cl/ <b>diamine</b> = 1-1.5/1.				

L9 ANSWER 17 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1994:192549 CAPLUS  
DN 120:192549  
TI Preparation of aromatic diisocyanates by **phosgenation** in gas phase  
IN Biskup, Klaus; Koenig, Christian; Waldau, Eckart  
PA Bayer A.-G., Germany  
SO Eur. Pat. Appl., 6 pp.

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CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 570799	A1	19931124	EP 1993-107560	19930510
	EP 570799	B1	19960619		
	R: BE, DE, FR, IT, NL				
	DE 4217019	A1	19931125	DE 1992-4217019	19920522
	CA 2096501	AA	19931123	CA 1993-2096501	19930518
	CA 2096501	C	20030729		
	BR 9302015	A	19931130	BR 1993-2015	19930520
	JP 06041046	A2	19940215	JP 1993-139885	19930520
	JP 3219903	B2	20011015		
PRAI	DE 1992-4217019	A	19920522		

OS MARPAT 120:192549

AB Continuous **phosgenation** of arom diamines, e.g., a mixture of 2,4- and 2,6-diaminotoluenes or bis(4-aminophenyl)methane, in the gas phase with residence time 0.5-5 s gives high yields (e.g., >99%) of diisocyanates without deposition of solid material in the reactor.

L9 ANSWER 18 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1991:448872 CAPLUS

DN 115:48872

TI Preparation of aliphatic isocyanates by reaction of aliphatic amines with **phosgene** in esters as solvents

IN Nagata, Teruyuki; Wada, Masaru; Mizuta, Hideki

PA Mitsui Toatsu Chemicals, Inc., Japan

SO Eur. Pat. Appl., 8 pp.

CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 424836	A1	19910502	EP 1990-120195	19901022
	EP 424836	B1	19950315		
	R: BE, DE, FR, GB, IT, NL				
	JP 03204851	A2	19910906	JP 1990-273283	19901015
	JP 2986888	B2	19991206		
	CA 2028162	AA	19910424	CA 1990-2028162	19901022
	US 5136086	A	19920804	US 1992-819657	19920113
PRAI	JP 1989-273811		19891023		
	JP 1989-275047		19891024		
	US 1990-598113		19901016		

OS CASREACT 115:48872

AB A process for the preparation of aliphatic polyisocyanates comprises the treatment

of an aliphatic polyamine or a hydrochloride thereof with COCl<sub>2</sub> in the presence of an ester as a solvent. A flask was charged with hexamethylenediamine (46.5 g) and MeCO<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>Me as a solvent (613.5 g) and 35 g HCl were blown over the mixture which was then heated to 150° and then COCl<sub>2</sub> was blown over the mix. at 29.4 g/h for 15 h; the yield of hexamethylene **diisocyanate** was 90%. The use of other solvents, e.g., mesitylene, gave a lower yield of hexamethylene **diisocyanate** and a higher yield of 6-chlorohexane **diisocyanate**.

L9 ANSWER 19 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1989:231172 CAPLUS

DN 110:231172

TI Procedure for the production of (cyclo)aliphatic diisocyanates

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IN Frosch, Hans Georg; Grave, Heinrich; Stutz, Herbert; Waldau, Eckart;  
Fuhrmann, Peter  
PA Bayer A.-G., Fed. Rep. Ger.  
SO Ger. Offen., 4 pp.  
CODEN: GWXXBX

DT Patent  
LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3714439	A1	19881110	DE 1987-3714439	19870430
	EP 289840	A1	19881109	EP 1988-106111	19880416
	EP 289840	B1	19901017		
	R: BE, DE, ES, FR, GB, IT, NL				
	US 4847408	A	19890711	US 1988-185721	19880425
	CA 1305165	A1	19920714	CA 1988-565025	19880425
	JP 63280050	A2	19881117	JP 1988-104461	19880428
	JP 08025984	B4	19960313		

PRAI DE 1987-3714439 19870430

OS CASREACT 110:231172; MARPAT 110:231172

AB A procedure for the preparation of OCN<sup>n</sup>RNCO [R = C1-15(cyclo)aliphatic hydrocarbon

moiety] by **phosgenation** of the corresponding H<sub>2</sub>NRNH<sub>2</sub> in the gas phase was characterized in that one: a) brings the gaseous **diamine**, optionally diluted with an inert gas or the vapors of an inert solvent, and COCl<sub>2</sub>, sep. heated to 200-600°, into reaction with each other in a cylindrical chamber at 200-600° without moving parts with the maintenance of a turbulent streaming into the reactor chamber; b) leads the gas mixture which continuously leaves the reaction chamber through an inert solvent which is kept at a temperature above the decomposition

temperature of the

carbamoyl chloride corresponding to the **diamine**; and c) subjects the **diisocyanate** dissolved in the inert solvent to a distillative work-up. In this manner, COCl<sub>2</sub> and H<sub>2</sub>N(CH<sub>2</sub>)<sub>6</sub>NH<sub>2</sub> reacted at 400° to give 98.0% OCN(CH<sub>2</sub>)<sub>6</sub>NCO.

L9 ANSWER 20 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1988:591412 CAPLUS

DN 109:191412

TI Preparation of aromatic diisocyanates and their mixtures for use in polyurethanes

IN Koenig, Klaus; Heitkaemper, Peter

PA Bayer A.-G., Fed. Rep. Ger.

SO Ger. Offen., 10 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3641702	A1	19880616	DE 1986-3641702	19861206
	EP 270906	A1	19880615	EP 1987-117283	19871124
	EP 270906	B1	19900221		

R: BE, DE, ES, FR, GB, IT, NL, SE

CA 1289146 A1 19910917 CA 1987-553105 19871130

US 4888125 A 19891219 US 1987-128295 19871203

JP 63159360 A2 19880702 JP 1987-306093 19871204

PRAI DE 1986-3641702 19861206

AB Mixts. of the diisocyanates p-OCNC<sub>6</sub>H<sub>4</sub>O(CH<sub>2</sub>CH<sub>2</sub>)nOC<sub>6</sub>H<sub>3</sub>(Me)NCO-2,1 30-100, p-OCNC<sub>6</sub>H<sub>4</sub>O(CH<sub>2</sub>CH<sub>2</sub>)nOC<sub>6</sub>H<sub>4</sub>NCO-p 0-50, and 2,1-OCN(Me)C<sub>6</sub>H<sub>3</sub>O(CH<sub>2</sub>CH<sub>2</sub>)nOC<sub>6</sub>H<sub>3</sub>(Me)NCO-2,1 0-50% (n = 1-3), useful in the manufacture of high-quality polyurethanes, are prepared by phosgenating the corresponding diamines.

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Adding 1348 g 2-(4-nitrophenoxy)ethyl p-toluenesulfonate in portions to 612 g 3-methyl-4-nitrophenol and 760 g 30% NaOMe in 6 L ethylene glycol at 80° and stirring 6 h at 100° gave 1029 g 1-(3-methyl-4-nitrophenoxy)-2-(4-nitrophenoxy)ethane, hydrogenation of which in DMF over Raney Ni at 75-90°/40-50 bar gave 92% **diamine**, **phosgenation** of which in PhCl gave 96% **diisocyanate** (m.p. 86-87°) containing 70 ppm hydrolyzable Cl.

L9 ANSWER 21 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1988:474090 CAPLUS  
 DN 109:74090  
 TI Preparation of a **diisocyanate** for use in plastics  
 IN Schmidt, Manfred; Koenig, Klaus; Heitkaemper, Peter; Pedain, Josef  
 PA Bayer A.-G., Fed. Rep. Ger.  
 SO Ger. Offen., 6 pp.  
 CODEN: GWXXBX  
 DT Patent  
 LA German  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3633712	A1	19880414	DE 1986-3633712	19861003
	CA 1296350	A1	19920225	CA 1987-547055	19870916
	EP 265668	A1	19880504	EP 1987-113871	19870923
	EP 265668	B1	19910109		
	R: BE, DE, FR, GB, IT, NL				
	JP 63091355	A2	19880422	JP 1987-245961	19871001
	US 5130466	A	19920714	US 1990-463713	19900111
PRAI	DE 1986-3633712		19861003		
	US 1987-103081		19870930		

AB The **diisocyanate** OCN(CH<sub>2</sub>)<sub>5</sub>CO<sub>2</sub>CH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>NCO (I) is prepared by **phosgenation** of the corresponding **diamine-2HCl** (II). Adding 930 g H<sub>2</sub>N(CH<sub>2</sub>)<sub>5</sub>COCl-HCl in 3.3 L CH<sub>2</sub>Cl<sub>2</sub> over 2 h to a HCl-saturated solution of 535 g H<sub>2</sub>NCH<sub>2</sub>C(Me)<sub>2</sub>CH<sub>2</sub>OH in 5 L CH<sub>2</sub>Cl<sub>2</sub> at 20° and heating at 40° gave 1420 g II. Passing COCl<sub>2</sub> at 50 g/h into a refluxing mixture of 289 g II and 3.5 L PhCl for 8 h gave 252 g I.

L9 ANSWER 22 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1988:455421 CAPLUS  
 DN 109:55421  
 TI Preparation of special diisocyanates for use in polyurethanes  
 IN Sanders, Josef  
 PA Bayer A.-G., Fed. Rep. Ger.  
 SO Ger. Offen., 11 pp.  
 CODEN: GWXXBX  
 DT Patent  
 LA German  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3628316	A1	19880225	DE 1986-3628316	19860821
	EP 257420	A1	19880302	EP 1987-111535	19870810
	EP 257420	B1	19900516		
	R: BE, DE, ES, FR, GB, IT, NL, SE				
	ES 2036198	T3	19930516	ES 1987-111535	19870810
	US 4820866	A	19890411	US 1987-84547	19870812
	CA 1292006	A1	19911112	CA 1987-544617	19870814
	JP 63057564	A2	19880312	JP 1987-205349	19870820
	US 4914238	A	19900403	US 1989-320678	19890308
PRAI	DE 1986-3628316		19860821		
	US 1987-84547		19870812		
OS	CASREACT 109:55421; MARPAT 109:55421				

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AB The title diisocyanates  $Z[OC_6H_3(R)NCO]_2$  [ $R = H, Me$ ;  $Z = (oxa)alkylene$ ] are prepared by the reaction of  $Z(OH)_2$  or  $O_2N(R)C_6H_3OZO$  with  $O_2N(R)C_6H_3Cl$ , reduction of  $NO_2$  groups, and **phosgenation**. Condensing 1 mol neopentyl glycol with 2.2 mol p- $O_2NC_6H_4Cl$  in DMSO containing powdered NaOH at 40-50° gave 92.8% 2,2-dimethyl-1,3-bis(4-nitrophenoxy)propane, hydrogenation of which in DMF over Raney Ni at 60°/50 bar gave 91% **diamine**. **Phosgenation** of the **diamine** in PhCl at -10° to 0° and refluxing gave 71.3% **diisocyanate** containing 0.007% hydrolyzable Cl.

L9 ANSWER 23 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1988:455417 CAPLUS

DN 109:55417

TI Aliphatic diisocyanates for preparation of polyurethanes

PA Bayer A.-G., Fed. Rep. Ger.

SO Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 62221661	A2	19870929	JP 1987-54317	19870311
	DE 3608354	A1	19870917	DE 1986-3608354	19860313
	DE 3620821	A1	19871223	DE 1986-3620821	19860621
	DE 3620821	C2	19940908		
PRAI	DE 1986-3608354		19860313		
	DE 1986-3620821		19860621		

OS CASREACT 109:55417

AB Diisocyanates  $OCNCR_1R_2ZCH_2NCO$  ( $R_1, R_2 = C_1-4$  alkyl;  $Z = C_2-9$  aliphatic hydrocarbylene) are prepared. Thus, a solution of  $COCl_2$  in PhCl was mixed with a solution of  $H_2N(CH_2)_3CMe_2NH_2$  at 10-20° for 1 h under vigorous stirring, heated to 127° during 3 h with addition of  $COCl_2$ , and refluxed 1 h at 127° to give 74%  $OCN(CH_2)_3CMe_2NCO$ .

L9 ANSWER 24 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1987:197297 CAPLUS

Correction of: 1986:34839

DN 106:197297

Correction of: 104:34839

TI Low-melting diphenylethane **diisocyanate** for polyurethanes

IN Kervennal, Jacques; Mathais, Henri

PA Atochem S. A., Fr.

SO Eur. Pat. Appl., 12 pp.

CODEN: EPXXDW

DT Patent

LA French

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 149388	A1	19850724	EP 1984-402584	19841213
	EP 149388	B1	19870812		
	R: BE, CH, DE, FR, GB, IT, LI, NL				
	FR 2557105	A1	19850628	FR 1983-20458	19831221
	FR 2557105	B1	19861128		
	JP 60202851	A2	19851014	JP 1984-254262	19841203
	US 4595744	A	19860617	US 1984-679571	19841207
PRAI	FR 1983-20458		19831221		

AB Diphenylethane **diisocyanate** (I), prepared by nitration of diphenylethane (II), hydrogenation, and **phosgenation**, is low-melting and useful in preparing polyurethanes. Thus, nitrating II with mixed acid, hydrogenating over Pd, and phosgenating gave I (2,4' 45, 4,4'

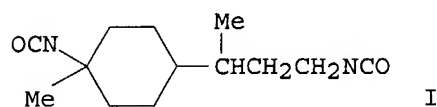
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30.3, 2,2' 15.0, 3,4' 5.0, and 2,3' 4.7%), m.p. 50°.

L9 ANSWER 25 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1987:6494 CAPLUS  
DN 106:6494  
TI Isocyanurate-containing polyisocyanates for polyurethane coatings  
IN Halpaap, Reinhard; Klein, Gerhard; Richter, Roland; Mueller, Hanns Peter;  
Pedain, Josef; Kreuder, Hans Joachim  
PA Bayer A.-G., Fed. Rep. Ger.  
SO Ger. Offen., 37 pp.  
CODEN: GWXXBX  
DT Patent  
LA German  
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3507719	A1	19860911	DE 1985-3507719	19850305
	CA 1286296	A1	19910716	CA 1986-501936	19860214
	EP 193828	A1	19860910	EP 1986-102373	19860224
	EP 193828	B1	19880427		
	R: AT, BE, CH, DE, FR, GB, IT, LI, NL, SE				
	AT 33844	E	19880515	AT 1986-102373	19860224
	JP 61212570	A2	19860920	JP 1986-45529	19860304
	JP 06045606	B4	19940615		
	ES 552631	A1	19880316	ES 1986-552631	19860304
	US 4851531	A	19890725	US 1987-74380	19870716
PRAI	DE 1985-3507719		19850305		
	US 1986-831210		19860220		
	EP 1986-102373		19860224		

GI

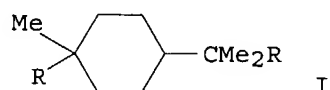


AB The title compns. are prepared without oligomeric trimer formation from aliphatic-cycloaliph. diisocyanates (20-50% NCO) having 1 sterically unhindered NCO group bonded to a primary C atom and 1 sterically hindered NCO group bonded to a tertiary C atom in the ring. The **diisocyanate I** was prepared by reductive amination of 3-(1-methyl-4-cyclohexenyl)butyraldehyde, hydrocyanation, hydrolysis, and **phosgenation** of the **diamine**. Stirring I 250, crown ether KOAc complex 1.0, and PhMe 250 g at 80° for 4 h and refluxing for 15 min gave a monoisocyanurate containing 17.6% NCO and <0.3% free I.

L9 ANSWER 26 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1984:68763 CAPLUS  
DN 100:68763  
TI Polymer intermediates from limonene  
AU Klein, Gerhard  
CS Bayer A.-G., Leverkusen, 5090, Fed. Rep. Ger.  
SO Forschungsber. - Bundesminist. Forsch. Technol., Technol. Forsch. Entwickl. (1983), BMFT-FB-T 83-053, 16 pp.  
CODEN: BFTEAJ; ISSN: 0340-7608  
DT Report  
LA German  
GI



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AB Limonene [138-86-3] was treated with HCN [74-90-8] to prepare I (R = NHCHO) [86178-67-8] which was treated with COCl<sub>2</sub> [75-44-5] to prepare I (R = isocyano) [86469-91-2]. Thermal isomerization gave I (R = cyano) [86469-91-2] which was hydrogenated to prepare I (R = CH<sub>2</sub>NH<sub>2</sub>) (II) [86469-92-3]. II was treated with COCl<sub>2</sub> to prepare I (R = CH<sub>2</sub>NCO) (III) [86469-93-4]. A film of polyamide [88666-91-5] prepared from II, isophthalic acid, and caprolactam was laminated with a polyethylene [9002-88-4] film, giving a composite film having better transparency and antiblocking properties, compared with a similar composite containing a polyamide prepared with isophoronediamine instead of II. An adduct of III and caprolactam was useful as a hardener for a saturated polyester containing

OH groups. III was useful as a substitute for isophorone diisocyanate in the preparation of polyurethanes.

L9 ANSWER 27 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1980:147777 CAPLUS

DN 92:147777

TI Aliphatic diisocyanates

IN Disteldorf, Josef; Huebel, Werner; Reiffer, Johannes; Kriebel, Guenter

PA Chemische Werke Huels A.-G., Fed. Rep. Ger.

SO Ger. Offen., 14 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 2830243	A1	19800124	DE 1978-2830243	19780710
	DE 2830243	C2	19870716		
	JP 55033465	A2	19800308	JP 1979-86545	19790710
PRAI	DE 1978-2830243		19780710		

AB 1,9-Diisocyanato-5-methylnonane (I) [68882-57-5] and 1,8-diisocyanato-2,4-dimethyloctane (II) [68882-56-4] are prepared (sep. or together) by the **phosgenation** (at -10 to +40° and then at 90-180°) of the hydrogenated reaction products of isobutene and acrylonitrile. Thus, 172 g 88:12 (weight) mixture of 1,9-diamino-5-methylnonane [45024-21-3] and 1,8-diamino-2,4-dimethyloctane [51121-88-1] in 2 L PhCl was saturated at 90° with CO<sub>2</sub> and then treated at 10° with 250 g COCl<sub>2</sub> [75-44-5] in PhCl over 1.5 h. After heating the product to 80° and then 133° with addition of more COCl<sub>2</sub>, a clear solution was obtained, giving I and II.

L9 ANSWER 28 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1976:179220 CAPLUS

DN 84:179220

TI Kinetics of the reaction of crystals of toluene-2,4-diamine and 4,4'-diphenylmethanediamine dihydrochlorides with **phosgene** dissolved in chlorobenzene

AU Konstantinov, I. I.; Kormushechkina, A. I.

CS USSR

SO Zhurnal Prikladnoi Khimii (Sankt-Peterburg, Russian Federation) (1976), 49(3), 596-9

CODEN: ZPKHAB; ISSN: 0044-4618

DT Journal

10670409-isocyanate

LA Russian

AB The rate-determining step of the title reactions was the formation of the 1st-stage solid product (aminoaryl isocyanate hydrochloride) on the crystal surface. The 2nd-stage product (**diisocyanate**) then dissolved in the PhCl. The activation energies with toluene-2,4-**diamine** and 4,4'-diphenylmethanediamine were 12.6 and 12.0 kcal/mole, resp.

L9 ANSWER 29 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1965:67227 CAPLUS

DN 62:67227

OREF 62:11989d-f

TI Polyurethans

IN Kaplan, Melvin

PA Allied Chemical Corp.

SO 12 pp.

DT Patent

LA Unavailable

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	FR 1375975		19640823	FR	
	GB 1007785			GB	
	NL 297969			NL	
	US 3215652		1965	US	

PRAI US 19620924

AB Polyether-polyols are treated with tolylene **diamine** (I) **phosgenation** products (amine equivalent 98-120), NCO-OH equivalent ratio 1.03: 1-1.05: 1, in the presence of a mixture of a blowing agent, tertiary amine, organotin compound, and a siloxane-oxyalkylene block copolymer to give rigid foams which are weak heat conductors. Thus, a solution (90%) of 244 parts I in 880 parts PhCl is added to a mixture (-10%) of 560 parts COCl<sub>2</sub> and 120 parts PhCl at a maximum of 20°. The mixture is heated to 136°, a HCl-COCl<sub>2</sub>-PhCl mixture is distilled, PhCl is distilled at 25 mm., and the residue is distilled at 10-12 mm. to give 70 parts tolylene **diisocyanate** (II) and 280 parts residue (III) (amine equivalent 106.3, viscosity (25°) 61 cp., d<sub>25</sub> 1.26) containing 74% II. A mixture of Actol 52-460 (OH number >460) 100, Bu<sub>2</sub>Sn dilaurate 0.37, Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH 1.0, Dow Corning 113 fluid 1.5, and FCCl<sub>3</sub> 38 parts is introduced into a mixer at 5.5 kg./min. at 23°, as III is introduced at 3.6 kg./min. at 22°, to give a mixture, NCO-OH ratio 1.03, which becomes hard after 6.5 min.; the product is molded at 41° to give a panel, d. 29 kg./m.<sup>3</sup>, containing 96.9% closed cells, and having a compression resistance of 1.3 kg./cm.<sup>2</sup> and a heat conductivity of 0.258 kcal./hr./m<sup>2</sup>/cm./°C.

L9 ANSWER 30 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1963:441342 CAPLUS

DN 59:41342

OREF 59:7403d-f

TI Preparation of tolylene **diisocyanate**. II. **Phosgenation** of tolylenediamine

AU Murakami, Tomohisa

CS Hodogaya Chem. Ind. Co., Yokohama, Japan

SO Yuki Gosei Kagaku Kyokaishi (1963), 21, 458-62

CODEN: YGKKAE; ISSN: 0037-9980

DT Journal

LA Unavailable

AB Reaction of diamines and COCl<sub>2</sub> at low temperature for conversion of the amino groups into a carbamoyl chloride and an amine-HCl group, and further reaction at high temperature with COCl<sub>2</sub> is known. When a **diamine** such

as tolylenediamine (I) is used, a high mol. weight urea compound is formed under the wrong reaction conditions and precipitates, so that the reaction with  $\text{COCl}_2$  becomes difficult, resulting in a considerable decrease in the reaction velocity and yield. For successful operation of this reaction, it is necessary to make sufficient contact of  $\text{COCl}_2$  with I in the low temperature reaction in order to decrease the amount of unreacted I in the product and give a sufficient supply of  $\text{COCl}_2$  in the high temperature reaction. It is desirable to use good solvents (e.g. ketone, esters, ethers) for the low temperature reaction product.  $\text{PhCl}$ ,  $\text{C}_6\text{H}_4\text{Cl}_2$ , etc., could be used as solvents by adding I solution to  $\text{COCl}_2$  solution with stirring, in order to give finely dispersed particles of the low temperature reaction product. The high temperature reaction is completed within 30 min. at  $130^\circ$  by sufficient introduction of  $\text{COCl}_2$  to the low temperature reaction product and the yield is high.

L9 ANSWER 31 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1950:679 CAPLUS

DN 44:679

OREF 44:109h-i,110b-i,111a-i,112a-i,113a-i,114a-i,115a-b

TI Polyurethans. IV. Mono- and polyisocyanates

AU Siefken, Werner

SO Ann. (1949), 562, 75-136

DT Journal

LA Unavailable

OS CASREACT 44:679

GI For diagram(s), see printed CA Issue.

AB cf. Bayer, C.A. 42, 6160c; Hebermehl, C.A. 43, 1604i. A comprehensive review of the researches in this field made by the staff of the former I. G. Farbenind., W. Altner, D. Delfs, A. Dierichs, E. Hartmann, E. Liese, A. Modersohn, S. Petersen, E. Prell, R. Putter, H. Rinke, W. Schulte, G. Schwaebel, H. Schwarz, G. Spielberger, K. Taube, A. Pielmann, K. Sigwart, H. Brock, J. Mierbach, E. Scholz, H. Glaser, F. Moller, and R. Schroter, including a brief literature survey with 62 refs. In general the  $\text{HCl}$  salts of amines were treated with  $\text{COCl}_2$  in excess, and the resultant  $\text{HCl}$  was rapidly removed by choosing a solvent in which the  $\text{RNCO}$ , but not  $\text{HCl}$  was soluble. Temps. were regulated to insure conversion of the intermediate  $\text{RNHCOC}_1$  into  $\text{RNCO}$ . In the aliphatic series, the yields of  $\text{RNCO}$  were uniformly satisfactory and arylaliph. amines, or alicyclic or heterocyclic amines, underwent very similar conversions. The reaction also applied to compds. of the type  $\text{RCH}(\text{NH}_2)\text{R}'$ . Solvents used included  $\text{PhMe}$ , xylene,  $\text{PhCl}$ ,  $\text{Cl}_2\text{C}_6\text{H}_4$ , and  $\text{C}_6\text{H}_3\text{Cl}_3$ , so chosen that the b.ps. of the solvent and the resulting  $\text{RNCO}$  showed a sufficient difference. The amine- $\text{HCl}$  may be dissolved in the solvent, or the free amine may be dissolved and then treated with dry  $\text{HCl}$ . Normally  $\text{COCl}_2$  was added until any insol.  $\text{HCl}$  salt was fully dissolved, the volatile gases then swept out by means of an inert gas, and the resulting products fractionated. Polymerization products were

retained as still residues; their amts. could be materially increased by the use of metallic catalysts (such as  $\text{FeCl}_3$ ,  $\text{SnCl}_4$ ,  $\text{ZnCl}_2$ , Fe carbonyl derivs., etc.). In individual cases,  $\text{HCl}$  is removed from  $\text{RNHCOC}_1$  by means of  $\text{Ca}(\text{OH})_2$  or derivs. of  $\text{CH}_2\text{O} \cdot \text{CH}_2$  (sometimes resulting in decreased yields of  $\text{RNCO}$ ). In special cases were used substituted ureas of the type  $\text{RR}'\text{NCONHR}''$  (formed from  $\text{RR}'\text{NCOCl} + \text{H}_2\text{NR}''$ ), where  $\text{R}''$  is a relatively small alkyl group and may be converted into  $\text{R}'\text{NCO}$  by heating above  $200^\circ$ . Another possibility was to treat compds. of the type  $2\text{-HOC}_6\text{H}_4\text{OCONHR}$  with the formation of  $\text{RNCO}$  and  $\text{o-C}_6\text{H}_4(\text{OH})_2$ . The conversions of amines containing such substituents as  $\text{Cl}$ ,  $\text{CN}$ ,  $\text{OR}'$ ,  $\text{CO}_2\text{R}'$ ,  $\text{COCl}$ , etc., into analogous isocyanates is discussed. Diamine, triamine, or tetramine  $\text{HCl}$  salts on  $\text{COCl}_2$  treatment may be converted into

analogous di-, tri-, and tetraisocyanates. Difficulties in the choice of suitable (large-scale) reactors are discussed. Whereas for an aliphatic RNCO, **phosgenation** may be carried out in stainless steel, aromatic di- and triamines require Pb-lined reactors. Batch or continuous **phosgenation** may be used. In determining the percentage NCO in an isocyanate, 2 methods were used: a cumbersome gravimetric method depending on the formation of a difficultly soluble Ph urea by condensation of RNCO with PhNH<sub>2</sub>, and the more practical procedure in which RNCO is treated with a known excess of Bu<sub>2</sub>NH in PhCl, thus giving rise very rapidly to Bu<sub>2</sub>NCONHR, and the unchanged Bu<sub>2</sub>NH is then titrated with HCl in the presence of MeOH (within 5 min. after inception of the quant. reaction). In the formation of 1,6-hexamethylene **diisocyanate** (I), b15 132° [bis(methylurethane), m. 113-14°], on repeated fractionation a small forerun was 6-chlorohexyl isocyanate (II), b12 108° [methylurethane (III), b10 150-2°; Cl(CH<sub>2</sub>)<sub>6</sub>NHCONH<sub>2</sub>, m. 128-9°]. [HCl.H<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>]<sub>2</sub> loses NH<sub>4</sub>Cl, forming CH<sub>2</sub>.(CH<sub>2</sub>)<sub>4</sub>.CH<sub>2</sub>.NH.HCl, which reacts with COCl<sub>2</sub> to give CH<sub>2</sub>.(CH<sub>2</sub>)<sub>4</sub>.CH<sub>2</sub>.NCOCl (isomeric with II), b11 116-18° (methylurethane, C<sub>6</sub>H<sub>12</sub>NCO<sub>2</sub>Me, b13 96-7°; and urea, C<sub>6</sub>H<sub>12</sub>NCONH<sub>2</sub>, m. 123-4°). III heated with PhONa formed PhOCH<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>NHCO<sub>2</sub>Me, which when heated with aqueous HCl-AcOH gave 6-phenoxyhexylamine-HCl, m. 142-3°. I in pseudocumene, heated 2 h. with pure COCl<sub>2</sub> at 160-65°, was not converted into II. On the other hand mixts. of HCl (gas) and COCl<sub>2</sub> acting on I gave small amts. of II after 48 h. **phosgenation**. The course of the reaction is discussed. Possibly Cl<sub>2</sub>CN(CH<sub>2</sub>)<sub>6</sub>NCO is first formed from I and then split into ClCN and II. MeO(CH<sub>2</sub>)<sub>3</sub>NH<sub>2</sub>.HCl treated 7 h. in 1-Cl<sub>10</sub>H<sub>7</sub>Cl at 140-50° with COCl<sub>2</sub> with stirring gave 81% Cl(CH<sub>2</sub>)<sub>3</sub>NCO, b16 54.6-4.8°, together with smaller amts. of (ClCH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, b1630°, which with PhONa yielded (PhOCH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, m. 61° (from EtOH). NC(CH<sub>2</sub>)<sub>5</sub>NH<sub>2</sub>, b16 118-19°, treated in PhCl with HCl, followed by COCl<sub>2</sub> at 120-30°, gave 94% NC(CH<sub>2</sub>)<sub>5</sub>NCO, b12 134-5° [NC(CH<sub>2</sub>)<sub>5</sub>NHCONH<sub>2</sub>, m. 142° (from EtOH)]. CH<sub>2</sub>(NH<sub>2</sub>.HCl)CO<sub>2</sub>Et in PhMe with COCl<sub>2</sub> gave 84.5% OCNCH<sub>2</sub>CO<sub>2</sub>Et, b11 67-8°, which with PhNH<sub>2</sub> in Et<sub>2</sub>O yielded PhNHCONHCH<sub>2</sub>CO<sub>2</sub>Et, m. 111° (from EtOH). From BuO(CH<sub>2</sub>)<sub>3</sub>NH<sub>2</sub>.HCl in PhMe was formed BuO(CH<sub>2</sub>)<sub>3</sub>NCO, b11 76-8°, and PhNHCONH(CH<sub>2</sub>)<sub>3</sub>OBu, m. 59-60° (from Et<sub>2</sub>O). By heating Ph<sub>2</sub>NCONHMe at 240-90°, 1 mol. MeNCO, b. 38-40°, and 1 mol. Ph<sub>2</sub>NH were formed. 2-HOC<sub>6</sub>H<sub>4</sub>O<sub>2</sub>CNH<sub>2</sub>Et, m. 142-4°, at 210-50° yielded EtNCO, b. 58-60°, and o-(HO)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>. In 1-Cl<sub>10</sub>H<sub>7</sub>Cl at 150° (PhNH)<sub>2</sub>CO with COCl<sub>2</sub> gave PhNCO, b16 55-7°. In C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub>, 3-HOCH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl and COCl<sub>2</sub> gave 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NCO (V), b11 118-19°; 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NHCO<sub>2</sub>Me, m. 87-8°, with pyridine yields 1-[3-(carbomethoxyamino)benzyl]pyridinium chloride, C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>N<sub>2</sub>Cl, m. 223-4°. Similarly, 3-MeOCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl (VI) on **phosgenation** gave 96% 3-MeOCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NCO, b11 108°, and 4% V; 3-MeOCH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>NHCONH<sub>2</sub>, m. 122-4°. VI treated in C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> at 140° with HCl prior to **phosgenation** gives 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>-NH<sub>2</sub>.HCl, converted readily into V. 3,3'-Bianisidine HCl salt in PhCl and COCl<sub>2</sub> at 150-60° gave after vacuum distillation at 150° a high yield of 3,3'-dimethoxy-4,4'-biphenylene **diisocyanate** (VII), m. 121-2°, forming a complex, 2VII.PhCl, m. 125-6°. The bis(methylurethane) of VII m. 215-16°. The following are examples of **phosgenation** of free bases. When 800 g. COCl<sub>2</sub> in 2 l. o-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub> in an ice-salt bath was treated with 200 g. molten (4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub> so that a temperature of about 0° was maintained, and the suspension was then heated to 130°, 215 g. (4-OCNC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub>, b0.1 156-8°, was formed [bis(methylurethane), C<sub>17</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>, m. 183-3.5°]. Similarly in PhCl, 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Me with COCl<sub>2</sub> gave 4-OCNC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Me, b11 122-4°, m. 49° (methylurethane, m. 177-8°), and tetrahydro-1,5-naphthylenediamine in C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> gave a mixture of tetrahydro-1,5-naphthylene **diisocyanate**, b0.09 129-32°, and a smaller amount of

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7,8-dihydro-1-naphthyl isocyanate, b15 137-8° [methylurethane, m. 86-7°; phenylurea (VIII), m. 210-12°], which was shown not to be identical with 1-naphthyl isocyanate, b12 140-2° (methylurethane, m. 122-3°; phenylurea, m. 220-2°), or with tetrahydro-ar-1-naphthyl isocyanate, b14 134-5° [methylurethane, m. 62-3°; phenylurea, m. 193-4°, also formed by hydrogenating VIII]. The following are examples of **phosgenation** of carbamic acids. 1,4-Diaminocyclohexane (345 g.) in 3 l. o-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub> was saturated at 90-95° with CO<sub>2</sub>, stirred 8 h., 700 g. COCl<sub>2</sub> introduced at 0°, the CO<sub>2</sub> removed, the mixture heated to 160°, and more COCl<sub>2</sub> added until after 14-16 h. the solution was clear; fractional distillation yielded a mixture of 1,4-cyclohexane diisocyanates, 1,4-(ONC)<sub>2</sub>C<sub>6</sub>H<sub>10</sub>, transform, m. 63-4° (from petr. ether) [bis(methylurethane), m. 264° (from MeOH)], and liquid cis form [characterized by its bis-(methylurethane), C<sub>10</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>, m. 139-40° (from Me<sub>2</sub>CO)]. Similarly, 4,4'-diaminodicyclohexylmethane gave 4,4'-(dicyclohexyl)methane **diisocyanate**, (4-OCNC<sub>6</sub>H<sub>10</sub>)<sub>2</sub>CH<sub>2</sub>, salvelike mass, b0.5-0.6 165-80°. The following are examples of **phosgenation** in the vapor phase. A mixture of 450 g. COCl<sub>2</sub> and 245 g. PhNH<sub>2</sub> passed in 1 h. through a tube at 230-40° while 130 g. 1-Cl<sub>10</sub>H<sub>7</sub>Cl was dropped in, gave 86% PhNCO. By entraining 80 g. p-(H<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>4</sub> with 35 l. CO<sub>2</sub> at 270-80° per h., heating the mixture to 470°, introducing 470 g. COCl<sub>2</sub>/h., and condensing in PhCl, S. obtained p-C<sub>6</sub>H<sub>4</sub>(NCO)<sub>2</sub>, m. 93-4° (after sublimation) [bis(methylurethane), m. 207°]. To 86.7 g. 3-HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl in 2 l. H<sub>2</sub>O containing 30 cc. HCl was added 42 cc. CCl<sub>4</sub>, thus forming 67 g. 3-HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>NCS, m. 165° (from MeOH); acid chloride, b14 152-4°, m. 22-3°, 40 g. of which in 200 cc. PhCl refluxed with 16 g. powdered NaN<sub>3</sub> gave 32 g. 3-OCNC<sub>6</sub>H<sub>4</sub>CNS, b14 140-2°, f.p. 4-6°. The following other isocyanates, RNCO, and their derivs. were prepared by methods analogous to those outlined. Most of these are new, but no differentiation has been made between new compds. and products previously prepared. In all cases (uncor.) b.p. or m.ps. were checked and some of the compds. were obviously impure. R = CH<sub>2</sub>:CHCH<sub>2</sub>, b. 87-9°; Pr, b. 88°; Bu, b. 114-16° (corresponding phenylurea, m. 129-30°); Me<sub>2</sub>CHCH<sub>2</sub>, b. 104-5° (phenylurea, m. 151-2°); Me<sub>3</sub>C, b. 85° (phenylurea, m. 167-8°). Am, b. 136-7° (AmNHCONHPh, m. 92-3°); isohexyl, b14 46°; BuCH<sub>2</sub>CH<sub>2</sub>, b15 75-9°; dodecyl, b11 140-6°; tetradecyl, b14 165-70°; hexadecyl, b14 186-8°; oleyl, b0.05-0.06 135-40°; octadecyl, b11 190-210°; CH<sub>2</sub>.CH<sub>2</sub>.SO<sub>2</sub>.CH<sub>2</sub>.CH (not characterized) (phenylurea, m. 190°); cyclohexyl, b11 54°; 2-decahydronaphthyl, b12 116-17°; C<sub>6</sub>H<sub>11</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>10</sub>, b0.25 120-3°; MeEtCH, b. 101-2° (phenylurea, m. 154°); iso-PrCHMe, b. 118-20° (phenylurea, m. 143-4°); Et<sub>2</sub>CH, b. 125-6° (phenylurea, m. 177-8°); Pr<sub>2</sub>CH, b13 58-9°; PhCHMe, b13 90-4°; PhCH<sub>2</sub>CH<sub>2</sub>CHMe, b12 117°; (C<sub>6</sub>H<sub>13</sub>)<sub>2</sub>CH, b12 139-41°; PrCH(C<sub>11</sub>H<sub>23</sub>), b12 150-70°; C<sub>17</sub>H<sub>35</sub>CHMe, b0.1 140-60°; ClCH<sub>2</sub>CH<sub>2</sub>, b16 42° [(ClCH<sub>2</sub>CH<sub>2</sub>NH)<sub>2</sub>CO, m. 128-9°]; Cl(CH<sub>2</sub>)<sub>4</sub>, b11 70-1° (phenylurea, m. 107°); Cl(CH<sub>2</sub>)<sub>5</sub>, b13 100° (methylurethane, m. 44°); Cl(CH<sub>2</sub>)<sub>7</sub>, b13 111°; Cl(CH<sub>2</sub>)<sub>4</sub>O(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, b0.1 76°; NC(CH<sub>2</sub>)<sub>2</sub>, b12 104°; [NC(CH<sub>2</sub>)<sub>2</sub>NH<sub>2</sub>CO, m. 148°]; NC(CH<sub>2</sub>)<sub>3</sub>, b10 103°; [NC(CH<sub>2</sub>)<sub>3</sub>NHCONH<sub>2</sub>, m. 142°]; MeO<sub>2</sub>C(CH<sub>2</sub>)<sub>5</sub>, b14 118°; MeO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 79-80°); EtO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 66°); PrO(CH<sub>2</sub>)<sub>3</sub>, b11 60-1° (phenylurea, m. 62°); iso-Pr(CH<sub>2</sub>)<sub>3</sub> (impure); BuO(CH<sub>2</sub>)<sub>3</sub>, b11 76-8° (phenylurea, m. 59-60°); iso-BuO(CH<sub>2</sub>)<sub>3</sub>, b14 78° (phenylurea, m. 80°); sec-BuO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 53-4°); cyclohexyloxypropyl, b11 113-14° (phenylurea, m. 100°); BuOCH<sub>2</sub>CH<sub>2</sub>O(CH<sub>2</sub>)<sub>3</sub>, b11 123-4° (phenylurea, liquid); C<sub>8</sub>H<sub>17</sub>O(CH<sub>2</sub>)<sub>3</sub>, b11

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135° (phenylurea, m. 55-6°); iso-C<sub>8</sub>H<sub>17</sub>, isomer, b<sub>11</sub> 125-8°; C<sub>10</sub>H<sub>21</sub>O(CH<sub>2</sub>)<sub>3</sub>, b<sub>11</sub> 160-2° (phenylurea, m. 58-9°); 3-(iso-octylcyclohexyloxy)propyl, b<sub>0.1-0.2</sub> 140-52°; o-tolyl, b<sub>9.5</sub> 63.2-3.4°; m-tolyl, b<sub>9.5</sub> 65.7-66.3°; p-tolyl, b<sub>10</sub> 67.6-67.8°; benzyl, b<sub>10</sub> 82-4° (phenylurea, m. 167-8°); xylyl (tech.), b<sub>12</sub> 78-80°; phenethyl, b<sub>10</sub> 98-100° (urea, m. 112°); 2,4,5-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, b<sub>12</sub> 100-1°; 2,4,6-isomer, b<sub>11</sub> 96-7°; Ph(CH<sub>2</sub>)<sub>3</sub>, b<sub>12</sub> 115°; 4-cyclohexylphenyl, b<sub>4</sub> 128-30°; 4-PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, b<sub>0.07</sub> 120° (methylurethane, m. 78-9°; urea, m. 158-9°); 2-methyl-4-cyclohexylphenyl, b<sub>4.5</sub> 138-42°; 3,4,6-Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, b<sub>11</sub> 124-6°, m. 60-1°; 3, 4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, b<sub>12</sub> 111-12°, m. 45° (methylurethane, m. 111-12°; urea, m. 153-4°); 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, b<sub>17</sub> 135°, m. 40-1°; 3-isomer, b<sub>11</sub> 130-1°, m. 50-1°; 4-isomer, b<sub>11</sub> 137-8°, m. 57°; 2-ClC<sub>6</sub>H<sub>4</sub>, b<sub>10</sub> 83.5°; 3-isomer, b<sub>10.5</sub> 83-6°; 4-isomer, b<sub>9.5</sub> 80.6-0.9°, m. 31-2° (methylurethane, m. 115°); 3-FO<sub>2</sub>SC<sub>6</sub>H<sub>4</sub>, b<sub>31-2</sub> 154-6°, m. 25° (methylurethane, m. 93°); 3-NCC<sub>6</sub>H<sub>4</sub>, b<sub>12</sub> 123°, m. 50-1° (methylurethane, m. 97-8°); 4,3-Cl(F<sub>3</sub>C)C<sub>6</sub>H<sub>3</sub>, b<sub>14</sub> 86-9°; 3,4-HO<sub>2</sub>C(HO)C<sub>6</sub>H<sub>3</sub>, decompose (methylurethane, m. 221°); 2, 4-Me(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, b<sub>23</sub> 168°, m. 75-8°; 4, 3-Me-(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, b<sub>0.2</sub> 100-2°; 2-MeOC<sub>6</sub>H<sub>4</sub>, b<sub>10</sub> 94.8-5.5°; 3-isomer, b<sub>10</sub> 94.6-4.8°; 4-isomer, b<sub>9.5</sub> 47.6-7.8°; 2, 5-Me(SCN)C<sub>6</sub>H<sub>3</sub>, b<sub>16.5</sub> 164° (methylurethane, m. 136-7°); 4,3-Cl(MeO<sub>2</sub>CNH)C<sub>6</sub>H<sub>3</sub>, b<sub>11</sub> 116°, m. 80°; 4-MeO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>, b. 122-4°, m. 49° (methylurethane, m. 177-8°); 2,4,5-Me<sub>2</sub>(O<sub>2</sub>N)C<sub>6</sub>H<sub>2</sub>, b<sub>2</sub> 110-25°; 3-MeCHClC<sub>6</sub>H<sub>4</sub>, b<sub>11</sub> 116°; EtOC<sub>6</sub>H<sub>4</sub>, b<sub>12</sub> 104-6°; 2, 5-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, b<sub>26-7</sub> 152-4°; 4-EtO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>, b<sub>17</sub> 144-8°, m. 30-2°; 3, 4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>, b<sub>14-15</sub> 176-8°; 4,3-Me(MeO<sub>2</sub>CNH)C<sub>6</sub>H<sub>3</sub>, m. 87°; 4,3-Me(EtO<sub>2</sub>CNH)C<sub>6</sub>H<sub>3</sub>, m. 72°; 4, 2-Cl(4-ClC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>3</sub>, m. 44-6°; 3-PhMeNSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 74-5°; 4-PhN:NC<sub>6</sub>H<sub>4</sub>, m. 97-8° (ethylurethane, m. 151°); 3, 4-Me(4-MeOC<sub>6</sub>H<sub>4</sub>N:N)C<sub>6</sub>H<sub>3</sub>, m. 80°; 5-methyl-2-methoxy-4-(2-chlorophenylazo)phenyl, m. 153°; 2-naphthyl, b<sub>14</sub> 144°, m. 56° (methylurethane, m. 113°); 5-nitro-1-naphthyl, m. 121-2° (methylurethane, m. 170°); 8-chloro-1-naphthyl, b<sub>0.1</sub> 125-7° (methylurethane, m. 119-20°); 1-chloro-2-naphthyl, b<sub>0.3</sub> 120°, m. 52-3° (methylurethane, m. 115-16°); tetrahydro-ac-2-naphthyl, b<sub>10</sub> 134-6° (phenylurea, m. 169-70°); decahydro-2-naphthyl, b<sub>12</sub> 116-17°; 3-phenanthryl, b<sub>4</sub> 198-204°, m. 48° (methylurethane, m. 140-2°); 4-(6-methyl-2-benzothiazolyl)phenyl, m. 143-4° (methylurethane, m. 203-5°); 9-ethyl-3-carbazolyl, b<sub>0.1</sub> 172-8°, m. 48° (methylurethane, m. 118-20°); 3-(9-carbazolyl)propyl, b<sub>7</sub> 240-2° (methylurethane, m. 107-9°); 3-pyrenyl, m. 92° (methylurethane, m. 203°); 12-chrysenyl, m. 155-6° (methylurethane, m. 204°); 9,10-ethanoanthr-11-ylmethyl, m. 92-4° (methylurethane, m.p. not given). The following diisocyanates (and derivs.) were prepared, a number of them by an azide degradation

method not discussed (R = -NCO): (CH<sub>2</sub>CH<sub>2</sub>R)<sub>2</sub>, b<sub>14</sub> 75-6°; R(CH<sub>2</sub>)<sub>3</sub>R, b<sub>14</sub> 86-7°; RCH<sub>2</sub>CH:CHCH<sub>2</sub>R (impure) [bis-(methylurethane), m. 137°]; R(CH<sub>2</sub>)<sub>4</sub>R, b<sub>14</sub> 102-4° [bis-(methylurethane), m. 129-30°]; (RCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>S, b<sub>0.5-1</sub> 100-20° {bis-urea [H<sub>2</sub>NCONH(CH<sub>2</sub>)<sub>2</sub>]<sub>2</sub>S, m. 210°}; R(CH<sub>2</sub>)<sub>5</sub>R, b<sub>15</sub> 123-5° [bis(methylurethane), m. 114-15°]; RCH<sub>2</sub>CHMeCH<sub>2</sub>CH<sub>2</sub>R, b<sub>9</sub> 98-9° (bis-urea, m. 181-2°); R(CH<sub>2</sub>)<sub>6</sub>R, b<sub>14</sub> 130-2° [bis(methylurethane), m. 113-14°]; [RCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>]<sub>2</sub>O, b<sub>14</sub> 137° (bis-urea, m. 141°); S-analog, b<sub>0.3-0.4</sub> 118°; R(CH<sub>2</sub>)<sub>7</sub>R, b<sub>14</sub> 140-2° [bis(methylurethane), m. 97.5-8°]; RCH<sub>2</sub>CMe<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>H<sub>2</sub>R, b<sub>12</sub> 120-2°, b<sub>16</sub> 132-4°; RCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH(OMe)(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>R, b<sub>12</sub>

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142-5°, (p-chlorophenylurea, m. 217-18°); R(CH<sub>2</sub>)<sub>8</sub>R, b<sub>11</sub>  
 146-8° [bis(methylurethane), m. 111-12°];  
 RCH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CHMeCH<sub>2</sub>R, b<sub>5</sub> 106° (bis-urea, m. 159-62°);  
 R(CH<sub>2</sub>)<sub>9</sub>R, b<sub>0.5</sub> 121° [bis(methylurethane), m. 102-5°];  
 R(CH<sub>2</sub>)<sub>10</sub>R, b<sub>0.8</sub> 128° [bis(methylurethane), m. 115°];  
 R(CH<sub>2</sub>)<sub>2</sub>CH(Obu)(CH<sub>2</sub>)<sub>3</sub>R, b<sub>2</sub> 142-55° (decomposition);  
 R(CH<sub>2</sub>)<sub>30</sub>(CH<sub>2</sub>)<sub>40</sub>(CH<sub>2</sub>)<sub>3</sub>R, b<sub>0.1</sub> 130-5°; R(CH<sub>2</sub>)<sub>11</sub>R, 0.06 124°  
 [bis(methylurethane), m. 97-8°]; R(CH<sub>2</sub>)<sub>12</sub>R, b<sub>0.08</sub> 135°  
 [bis(methylurethane), m. 117-18°]; [R(CH<sub>2</sub>)<sub>6</sub>]<sub>2</sub>S, b<sub>0.7-0.8</sub>  
 180-5° [bis(methylurethane), m. 87°]; m-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>R)<sub>2</sub>, b<sub>12</sub>  
 159-62°, [bis(methylurethane), m. 110-11°]; p-isomer, b<sub>16</sub>  
 172°, m. 45-6° [bis(methylurethane), m. 184°]; 1,  
 2-C<sub>6</sub>H<sub>10</sub>(CH<sub>2</sub>R)<sub>2</sub> (cyclohexane), b<sub>23-24</sub> 165-8°; 1,4-isomer, b<sub>11</sub>  
 154-6° [bis(methylurethane), m. 163°]; p-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>CH<sub>2</sub>R)<sub>2</sub>,  
 b<sub>0.1-0.2</sub> 142-5°; 1, 4-Cl<sub>10</sub>H<sub>6</sub>(CH<sub>2</sub>R)<sub>2</sub>, b<sub>1</sub> 183-4°  
 [bis(methylurethane), m. 167°]; 1, 5-isomer, m. 88-9°  
 [bis(methylurethane), m. 215-16°]; RCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CHMe<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>  
 3R, b<sub>4</sub> 165-72°; [C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>3</sub>R]<sub>2</sub>, m. 29° [bis(methylurethane),  
 m. 156-7°]; (IX), m. 235-7° (analogous dicyanate not  
 characterized). RCH<sub>2</sub>CH<sub>2</sub>CHR<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>, b<sub>14</sub> 120-30°; impure  
 CHMe<sub>2</sub>CHR<sub>2</sub>CH<sub>2</sub>CHR<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>, b<sub>12</sub> 127-9° Et homolog, b<sub>19</sub> 140-4°;  
 CH<sub>2</sub>(CH<sub>2</sub>CH<sub>2</sub>CHR<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>, b<sub>0.5-0.6</sub> 165-80°; MeCH(C<sub>6</sub>H<sub>10</sub>R)<sub>2</sub>  
 homolog, b<sub>4.5</sub> 198-208° [bis-(methylurethane), m. 182-3°];  
 Me<sub>2</sub>C(C<sub>6</sub>H<sub>10</sub>R)<sub>2</sub> homolog, b<sub>8-10</sub> 208-30°; (CH<sub>2</sub>CH<sub>2</sub>CHR<sub>2</sub>CH<sub>2</sub>CHMe<sub>2</sub>CH)<sub>2</sub>  
 CH<sub>2</sub>, b<sub>5</sub> 200-12°; 3,3'-dimethyl isomer, b<sub>9</sub> 198-210°;  
 3,3',5,5'-tetra-Me homolog, b<sub>7</sub> 205-12° [bis(methylurethane), m.  
 204-7°]; p-RCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b<sub>19</sub> 152°; p-RCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b<sub>15</sub>  
 156°; m-RCH(Me)C<sub>6</sub>H<sub>4</sub>R, b<sub>11</sub> 134° [bis(methylurethane),  
 m. 112-13°]; m-RCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b<sub>0.5</sub> 118-20°; p-isomer,  
 b<sub>0.5</sub> 100-115°; m-RCHMeCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b<sub>2</sub> 146-9°; p-isomer,  
 b<sub>3.5</sub> 142-5°; tetrahydro-1,5-naphthylene diisocyanate,  
 b<sub>0.04</sub> 117-18°, 4-(4-RC<sub>6</sub>H<sub>10</sub>)C<sub>6</sub>H<sub>4</sub>R (termed "4,4'-hexahydrobenzidine  
 derivative"), b<sub>0.7</sub> 155-60°, m. 38-44° [bis(methylurethane), m.  
 216° (poorly)]; 4-(4-RC<sub>6</sub>H<sub>10</sub>-CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>R, b<sub>0.2-0.3</sub>, m. 152-6°;  
 m-C<sub>6</sub>H<sub>4</sub>R<sub>2</sub> b<sub>12</sub>, 104-6°, m. 51° [bis(methylurethane), m.  
 157-7.5°]; p-isomer, b<sub>12</sub> 110-12°, m. 94-6°  
 [bis(methylurethane), m. 209-10°]; 1,2,4-MeC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>18</sub>  
 124-6°, m. 21° (33° also given) [bis(methylurethane),  
 m. 170-1°]; 1,2, 6-isomer, b<sub>18</sub> 129-33°; 1,2,5-isomer, b<sub>15</sub>  
 138-9°, m. 39°; 1,3,5-isomer, b<sub>17</sub> 133.5-4.5°;  
 1,3,2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b<sub>17</sub> 140-4°; 1,3,4,6-isomer, b<sub>15</sub> 139°, m.  
 70-1°; 1,4,2,5-isomer, b<sub>14</sub> 138-43°, m. 82° (also  
 given as 76°); 1,2,4-EtC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>12</sub> 124-6°;  
 1,2,4-iso-PrC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>12</sub> 136-40° [bis(methylurethane), m.  
 160-7°]; ??-Et<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub> b<sub>11</sub> 138-40°; ??-iso-Pr<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b<sub>12</sub>  
 148-60°; 1,2,4-ClC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>11</sub> 122-4°; 1,2,4-O<sub>2</sub>NC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>,  
 b<sub>14</sub> 160-80° [bis(methylurethane), m. 197-8°]; 1,2,5-isomer,  
 b<sub>4</sub> 165-73°, m. 59-61° [bis(methylurethane), m.  
 157-8°]; 1,3,4,6-Cl<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b<sub>12</sub> 140-6°; 1,3,2,4-isomer,  
 b<sub>1.5</sub> 120-6°, m. 64-5° [bis(methylurethane), m.  
 184-5°]; 1,4,2,5-isomer, m. 134-7°;  
 1,4,2,5-Cl(MeO)C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 134°; 1,2,4-MeOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, m. 75°;  
 1,2,5-isomer, m. 89°; 1,4,2,5-Me(MeO)C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 106-7°;  
 1,2,4-EtOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>16</sub> 162-4°, m. 56°; 1,3,4,6-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>  
 , m. 125° [bis-(methylurethane), m. 154°]; 1, 4, 2,  
 5-isomer, m. 180-1°; 1,2,4-PrOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b<sub>15</sub> 164-5°; 1-iso-BuO  
 homolog, b<sub>14</sub> 180-1°; 1,4,2,5-(EtO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 128°;  
 (p-RC<sub>6</sub>H<sub>4</sub>N)<sub>2</sub>, m. 158-61° [bis(methylurethane), m. 241-3°];  
 p-[4, 2-RMeC<sub>6</sub>H<sub>3</sub>N:N)C<sub>6</sub>H<sub>4</sub>R, m. 123-5° [bis(methylurethane), m.  
 244-8°]; 1, 4-(p-RC<sub>6</sub>H<sub>4</sub>N:N)C<sub>6</sub>H<sub>10</sub>R, m. 177-8°,  
 [bis(methylurethane), m. 210-12°]; p-(o-RC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>R, m.  
 60-2°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>O, b<sub>5</sub> 196°, m. 66-8°;

(o-RC<sub>6</sub>H<sub>4</sub>OCH<sub>2</sub>)<sub>2</sub> and (o-RC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>O (prepared but not characterized). The following diisocyanato derivs. of naphthalene were prepared: 1, 4, m. 67-70° [bis(methylurethane), m. 220-2°]; 1, 5, m. 130-2° (subliming in high vacuum) [bis(methylurethane), m. 245-6°]; 2, 6, m. 152-4° [bis(methylurethane), m. 244-6°]; 2, 7, m. 152-3°; (2, 1-RC<sub>10</sub>H<sub>6</sub>)<sub>2</sub>, m. 132-3° [bis(methylurethane), m. 185-7°]. The following diisocyanato derivs. of Ph<sub>2</sub> were formed: 2, 4', b0.5 140-50° [bis(methylurethane), m. 171-2°]; 4, 4' (X), b0.5 150-60° [bis(methylurethane), m. 244°]; 3, 3'-di-Me derivative of X, m. 68-9° [bis(methylurethane), m. 208°]; 3, 3'-di-MeO derivative of X, m. 121-2° [bis(methylurethane), m. 215-16°]; 2-NO<sub>2</sub> derivative of X, m. 119-21° [bis(methylurethane), m. 211-12°]. The following diisocyanates were formed: (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub>, b0.2 170°, m. 46° (also given as 37.6°) [bis(dimethylurethane), m. 183-3.5°]; (2, 4-MeRC<sub>6</sub>H<sub>3</sub>)<sub>2</sub>CH<sub>2</sub>, b5 208-11°, m. 65-7°; p-R(C<sub>6</sub>H<sub>4</sub>2CMe<sub>2</sub>, b5.6 212°, m. 91.5°; (2,5,4-Me<sub>2</sub>RC<sub>6</sub>H<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, m. 127.6°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>6</sub>11(?), b0.1 195-200° [bis(methylurethane), m. 173°]; [4, 3-R(MeO) C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 97-8°; [3, 4-R(MeO) C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 90-3°; [3, 4-R(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 74°; [4, 2, 5-RMe (MeO) C<sub>6</sub>H<sub>2</sub>]<sub>2</sub>CH<sub>2</sub>, m. 128-9°; [4, 3-RC<sub>1</sub>C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CMe<sub>2</sub>, m. 100-1°; (m-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub> CO, m. 118-20°; 1,2,4-PhCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b20 223-7° [bis(methylurethane), m. 154-5°]; m-(p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub> CHC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub> (not characterized) [mono- or bis(phenylurea), sinters about 145°]; p-isomer (not characterized) [mono- or bis(phenylurea) (?), m. 224-6°]; (4, 2, 5-RMe<sub>2</sub>C<sub>6</sub>H<sub>2</sub>)<sub>2</sub> CHPh, m. 116-18°. The following diisocyanato derivs. of fused rings and of aromatic S compds. were formed: 2, 7-R<sub>2</sub>-fluorene, m. 133°; 2, 6-R<sub>2</sub>-anthraquinone, m. 250-2°; 9-ethyl-2, 7-R<sub>2</sub>-carbazole (S. gives 3, 6), m. 122-3°; 3, 8-R<sub>2</sub>-pyrene, m. 234° [bis-(methylurethane), m. 264°]; 5, 11-R<sub>2</sub>-chrysene, m. 274° (S. gives 2, 8). The following diisocyanates were prepared from aromatic S-containing compds. 1,2,4-PhSC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, m. 73-4°; (p-RC<sub>6</sub>H<sub>4</sub> S)<sub>2</sub>, m. 58-60°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub> SO<sub>2</sub>, m. 154° [bis-(methylurethane), m. 262-4°]; XI, m. 166-8° [bis(methylurethane), m. 235-7°]; 1,2,4-(p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>) C<sub>6</sub> H<sub>3</sub>R<sub>2</sub> (impure); p-(3, 4-RMeC<sub>6</sub>H<sub>3</sub>SO<sub>3</sub>) C<sub>6</sub>H<sub>4</sub>R, m. 67.5°; 4-MeO analog, m. 104-6°; (5, 2-RMeC<sub>6</sub>H<sub>3</sub>S)<sub>2</sub>, m. 77-8°; 2, 5-isomer, m. 74-6°; 3, 4-isomer, m. 88°; [3, 4-R(MeO)-C<sub>6</sub>H<sub>3</sub>S]<sub>2</sub>, m. 66-7°; 4, 3-isomer, m. 101° [3, 4-R(MeO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> SO<sub>2</sub>, m. 165°; 1, 3, 4-(3, 4-RMeC<sub>6</sub> H<sub>3</sub>SO<sub>2</sub> NH)C<sub>6</sub>H<sub>3</sub>MeR, m. 191-6°; (p-RC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NHCH<sub>2</sub>)<sub>2</sub> (m. poorly 178°); [4, 3-R(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>]<sub>2</sub>S (not characterized); [3, 4-R(MeO)C<sub>6</sub>H<sub>3</sub>SCH<sub>2</sub>]<sub>2</sub>, m. 118°; [4, 3-R(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>]<sub>2</sub> SO<sub>2</sub> (not characterized); [3, 4-(MeO)C<sub>6</sub>H<sub>3</sub>SO<sub>2</sub>CH<sub>2</sub>]<sub>2</sub>, m. 265-7°; [3, 4-(MeO)C<sub>6</sub>H<sub>3</sub>SO<sub>2</sub>NHCH<sub>2</sub>]<sub>2</sub>, m. 192°. The following triisocyanates were formed: 1, 2-4, 6-MeC<sub>6</sub>H<sub>2</sub>R<sub>3</sub>, b1-3 133-9°, m. 75° [tris(methylurethane), m. 195°]; 1, 3, 5, 2, 4, 6-Me<sub>3</sub>C<sub>6</sub>R<sub>3</sub>, m. 93° [tris(methylurethane), m. 284°]; 1, 3, 7-C<sub>10</sub>H<sub>5</sub>R<sub>3</sub>, m. 162-3° [tris(methylurethane), m. 217°]; p-(2, 4-R<sub>2</sub>C<sub>6</sub>H<sub>3</sub>) C<sub>6</sub>H<sub>4</sub>R, b1.5-2 194-8°, m. 71°; p-(2, 4-R<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>R, undistillable; p-(2, 4, 3-R<sub>2</sub>MeC<sub>6</sub>H<sub>2</sub>CH<sub>2</sub>)C<sub>6</sub> H<sub>4</sub>R, b4 224°, m. 75-7° [tris(methylurethane), m. 167°]; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>CH, m. 89-90°. The following were also formed: (4, 2, 5-Me<sub>2</sub>R<sub>2</sub>C<sub>6</sub>H<sub>2</sub>)<sub>2</sub> CH<sub>2</sub>, m. 154-6°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NCOC<sub>1</sub>, b2-3 230-4°, m. 74-77° [bis(methylurethane), m. 182-4°]. The following dimers of the type formula [1,2,4-RC<sub>6</sub>H<sub>3</sub>(NCO)<sub>2</sub>]<sub>2</sub> were prepared: R = Cl, m. 177°; Me, m. 156°; Et, m. 148°; EtO, m. 185-6°; Me<sub>2</sub>CH, m. 125°; Me<sub>2</sub> CHO, m. 158°; iso-BuO, m. 176°. Also formed was [1,2,5-MeC<sub>6</sub>H<sub>3</sub>(NCO)<sub>2</sub>]<sub>2</sub>, m. 186°.



10670409-isocyanate

COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	99.06	99.27
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-21.48	-21.48

FILE 'USPATFULL' ENTERED AT 13:55:18 ON 21 JUN 2004  
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REVISED CLASS FIELDS (/NCL) LAST RELOADED: Apr 2004  
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Apr 2004

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This file contains CAS Registry Numbers for easy and accurate  
substance identification.

=> s phosgenation

L16 3991 PHOSGENATION

=> s phosgene

L17 19237 PHOSGENE

=> s l16 and l17

L18 1912 L16 AND L17

=> s l18 and diamine

68324 DIAMINE

L19 691 L18 AND DIAMINE

=> s l19 and isocyanate

59036 ISOCYANATE

L20 602 L19 AND ISOCYANATE

=> s l20 and spray

213596 SPRAY

10670409-isocyanate

L21 109 L20 AND SPRAY

=> s l21 and nozzles

107350 NOZZLES

L22 2 L21 AND NOZZLES

=> s "spray nozzles"

213596 "SPRAY"

107350 "NOZZLES"

L23 13823 "SPRAY NOZZLES"  
("SPRAY"(W) "NOZZLES")

=> s l23 and l20

L24 0 L23 AND L20

=> d 1-2 bib abs l22

L22 ANSWER 1 OF 2 USPATFULL on STN

AN 2003:282455 USPATFULL

TI Stabilized aqueous crosslinker dispersions

IN Rische, Thorsten, Unna, GERMANY, FEDERAL REPUBLIC OF  
Naujoks, Karin, Odenthal, GERMANY, FEDERAL REPUBLIC OF  
Meixner, Juergen, Krefeld, GERMANY, FEDERAL REPUBLIC OF  
Feller, Thomas, Solingen, GERMANY, FEDERAL REPUBLIC OF  
Konig, Eberhard, Leverkusen, GERMANY, FEDERAL REPUBLIC OF

PI US 2003198796 A1 20031023

AI US 2003-341977 A1 20030114 (10)

PRAI DE 2002-10201545 20020117

DT Utility

FS APPLICATION

LREP BAYER POLYMERS LLC, 100 BAYER ROAD, PITTSBURGH, PA, 15205

CLMN Number of Claims: 20

ECL Exemplary Claim: 1

DRWN No Drawings

LN.CNT 784

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB The present invention relates to a water-dispersible crosslinker composition containing

A) at least one hydrophilically-modified, blocked polyisocyanate,

B) at least one stabilizing agent containing

a) at least one amine containing a structural unit corresponding to formula (I) ##STR1##

which does not contain hydrazide groups,

b) at least one compound containing a structural unit corresponding to formula (II)

--CO--NH--NH-- (II)

and

c) optionally a stabilizing component other than a) and b), and

C) optionally an organic solvent.

The present invention also relates to an aqueous solution or dispersion containing this crosslinker composition, to aqueous coating compositions containing this crosslinker composition and to glass fibers coated with

10670409-isocyanate

this coating composition.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L22 ANSWER 2 OF 2 USPATFULL on STN

AN 76:40550 USPATFULL

TI Colored poly (urethane) urea powders

IN Weber, Karl-Arnold, Leverkusen, Germany, Federal Republic of  
Reiff, Helmut, New Martinsville, WV, United States

PA Dieterich, Dieter, Leverkusen, Germany, Federal Republic of  
Bayer Aktiengesellschaft, Leverkusen, Germany, Federal Republic of  
(non-U.S. corporation)

PI US 3970601 19760720

AI US 1975-573491 19750501 (5)

PRAI DE 1974-2425810 19740528

DT Utility

FS Granted

EXNAM Primary Examiner: Welsh, M. J.

LREP Pope, Lawrence S., Harsh, Gene

CLMN Number of Claims: 5

ECL Exemplary Claim: 1

DRWN No Drawings

LN.CNT 1198

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB The instant invention relates to thermoplastic polyurea powders,  
optionally containing urethane groups, characterized by

A. a smooth, substantially spherical or lenticular surface,

B. an average particle diameter of 5 to 1,000 $\mu$ , preferably 10 to  
200 $\mu$ ,

C. a urea group content, and, optionally, a urethane group content, of 8  
to 30% by weight,

D. an ionic group content of 0.01-0.15 milliequivalents per gram of  
solids, preferably 0.02-0.1 milliequivalent per gram and

E. 0.1 to 10% by weight, preferably 0.2 to 5% by weight, of an organic  
dye incorporated in the polymer molecule by way of at least one urethane  
and/or urea group,

And the process of manufacture thereof.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.